[redacted]

DEPARTMENT *of* CHEMICAL *and* BIOCHEMICAL ENGINEERING

Faculty of Engineering Science

The UNIVERSITY *of* WESTERN ONTARIO

PROCESSING OF CASHEW NUT SHELL LIQUID

TECHNICAL REPORT
PROJECT FILE: 89-1039-01

September 12, 1992

Issuing the Report:
Dr. H.I.de Lasa and S. Afara
Chemical Reactor Engineering Centre
Faculty of Engineering Science
University of Western Ontario
London, Ontario, Canada N6A 5B9
Tel: (519) 661-2144
Fax: (519) 661-3498

Circulation of the report at UWO:
Dr. M. Bergougnou
Professor

Circulation of the report at IDRC:
Dr. Pierre Zaya
Associate Director
International Development Research Centre
P.O.Box 8500,
Ottawa, Ontario
K1G 3H9

TABLE OF CONTENT

PROCESSING OF CASHEW NUT SHELL LIQUID / MOZAMBIQUE

TECHNICAL REPORT

Introduction:

The general objective of the present project is to develop a process to transform the raw CNSL obtained from the cashew treatment plants into chemical products useful for the mozambican market.

As stated in the original application, the detailed objectives can be summarized as follows:

1 - Preliminary study of the process.
Status: Achieved 100%
2 - Design of the pilot plant for the conversion of CNSL.
Status: Achieved 100%
3 - Construction of the pilot plant.
Status: Achieved 100%
4 - Trial testing of the pilot plant.
Status: Pending
5 - Optimization of the process.
Status: Pending

Preliminary Study of the Process:

The purpose of the preliminary study is to characterize the CNSL and to obtain some estimates of the product yield (light gases, phenol, hydrocarbon, tar) as a function of temperature and residence time. This phase was conducted at the Chemical Reactor Engineering Centre (CREC) by Mr. Radek Bragança, a University Eduardo Mondlane (UEM) researcher, during his stay in Canada from November 1990 to February 1991. In addition, Mr. Braganca was directly involved in the design aspects of the pilot plant and the proper procedure of its operation. In summary, the following points were analyzed under the direct supervision and guidance of Drs. Bergougnou and de Lasa.

1 - Cracking of the CNSL in a Microcatalytic Reactor.
2 - Study of the physical/chemical parameters of the process.
3 - Identification of the main reaction products by gas chromatography.
4 - Computer simulation of the industrial unit.

The work carried out in Canada allowed the pyrolysis of CNSL at higher temperatures (450°C to 750°C) and very short residence times (around 1 second). The following table is a summary of these results:

Product (wt%)	Low Temperature	High Temperature
Tar	0.20	Traces
Phenols	0.44	0.43
Hydrocarbons	0.30	0.37
Gaseous fraction	0.06	0.20

Hence, the following explanation was concluded by UEM researchers in a progress report addressed to CREC on September 11, 1991.

a) The amount of tar is significantly reduced at higher temperatures since the short residence time does not allow the polymerization of the CNSL and the reaction product as well. The absence of tar is desirable under the operation point of view: the emulsion formed by the colloidal tar particles would be difficult to separate and would require a constant cleaning of the reactor to avoid clogging.

b) Due to higher temperatures used, the amount of gases is increased as a consequence of the larger extent of the cracking reaction. Product gas can be used to feed diesel generators for electricity production inside the CNSL plant and in rural areas nearby. On the other hand, tar can be obtained in considerable amounts, if desirable, by heating raw CNSL for long periods.

c) Another advantage of working at high temperatures and short residence times, as outlined by UEM researchers, is to avoid extended polymerization of the reaction products. This gives, as well, the possibility of production of phenols with higher commercial value such as the m-cresol, vinyl phenols,...

In summary, as a first step toward the implementation of an industrial scale operation in Mozambique under the guidance of Canadian researchers, this pilot plant facility is a very crucial installation to UEM reserachers in order to confirm the laboratory results obtained in Canada through further experiments to optimize this process.

<u>Design of the pilot plant</u>:

This joint task was conducted with the collaboration of the Canadian team by the visiting researcher Mr. Radek Bragança. A computer simulation of the industrial unit was carried out using "HYSIM" package available at CREC. This simulation allowed a detailed study of several suitable processing possibilities, as well as, in acquiring a general conception of the approved pilot plant. Specific sizes and characteristics of the different units to be built in Canada were discussed and reviewed with Mr. Bob Kager, technical personnel, of the Mechanical Shop of the University of Western Ontario. In addition, contacts were established between Mr. Bragança and potential Canadian manufacturers and suppliers to furnish additional necessary support equipments related to the safe operation of the pilot plant in Mozambique.

The process was developed in such a way so as to keep it as simple as possible, at the same time flexible enough for testing a number of different operating conditions. The final flowsheet of the pilot plant as conceptualized by the UEM-CREC researchers involved in this project is outlined in Figure 1. It consists of 3 main sections:

1 - "Downer" where the CNSL is fed through special nozzle system and subjected to thermal cracking. Superheated steam is used as heat carrier and supplied by a set of 4 nozzles providing the heat for the endothermic cracking reaction.

2 - "Quencher" where water droplets impinge on the hot product stream, stopping the reaction by reducing drastically the temperature.

3 - "Product separation, condensation and sampling". The effluents from the hopper are brought to room temperature and respectively collected in vapour and liquid phases. Representative samples of these will be assessed by gas chromatography.

Description of the pilot plant

The CNSL, typically 1 kg/h, is pumped at room temperature from the reservoir R1 via a positive displacement pump P1 to the reactor R. A series of valves and graduated cylinder are installed before P1 to monitor and ensure proper feedstock flowrate during the experimental run. The top of the reactor R is fitted with a cooling jacket to prevent the precracking of CNSL prior to atomization through the nozzle. The reactor R is fabricated from Inconel to withstand high temperatures.

Superheated steam, typically 9 kg/h, generated by the boiler (Figure 2) provides the necessary heat for the cracking reaction. Steam is used as heat carrier. This stream is monitored and transported in the same way as the feedstock, through a series of valves and graduated cylinders to a manifold connected to a set of 4 nozzles. Water is pumped, by a positive displacement pump (P2) to the boiler and the exit temperature (T11) is recorded prior to being conveyed to the reactor. In the reactor, the superheated atomized steam flow impinge on the incoming atomized feedstock thus creating a turbulent well mixed zone.

The fast pyrolysis of CNSL is conducted in a "downflow" reactor with a pattern close to plug flow regime. The CNSL and steam are intermixed in the reactor head (Figure 3) through a highly efficient CNSL-Steam nozzle injector system, the entrained downwards through a 3 m long Inconel pipe fitted with a series of 6 thermocouples, equally spaced and used to monitor the temperature of the reaction along the reactor body. A General view of the reactor and thermocouples inserts is presented in Figure 1.

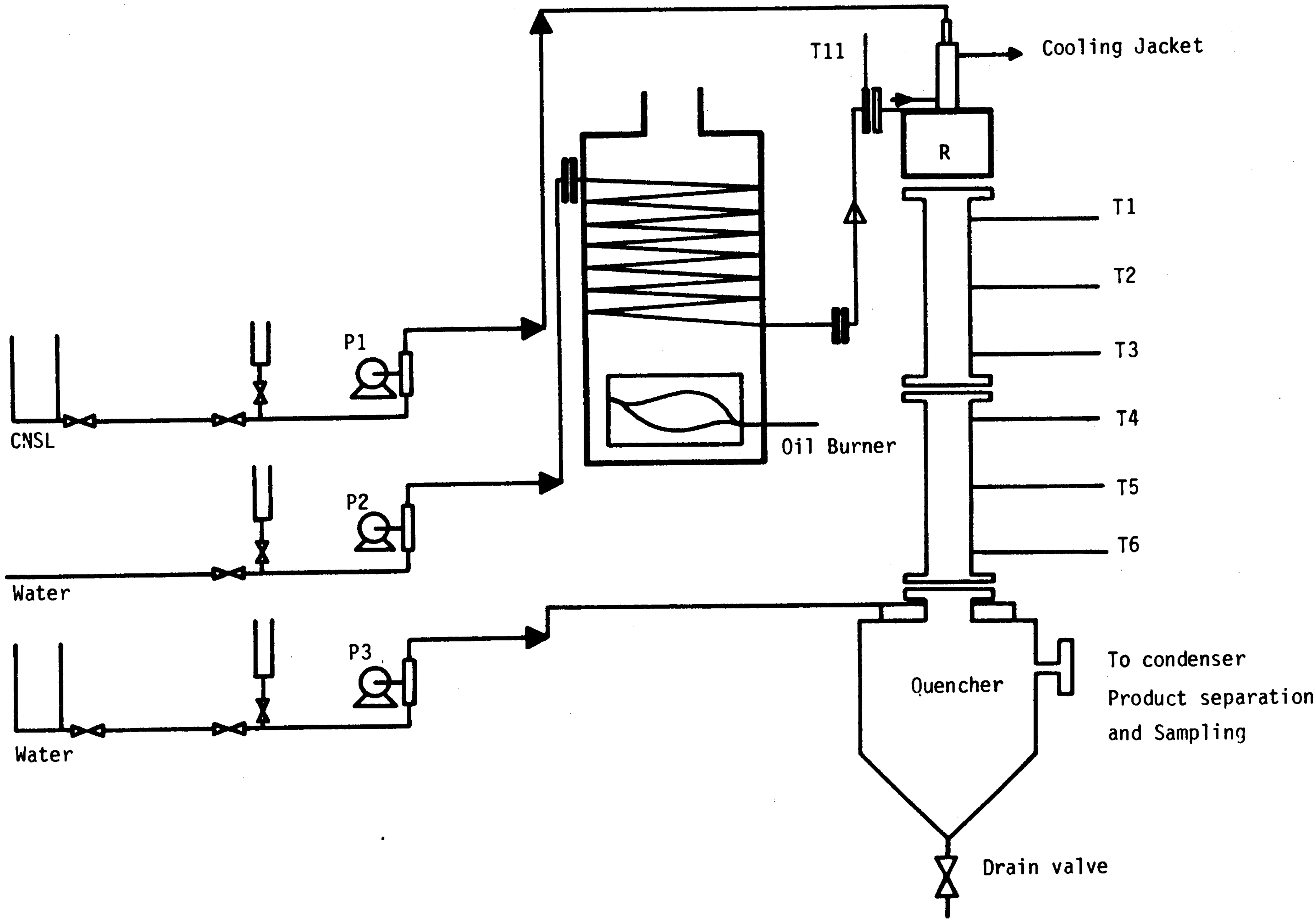
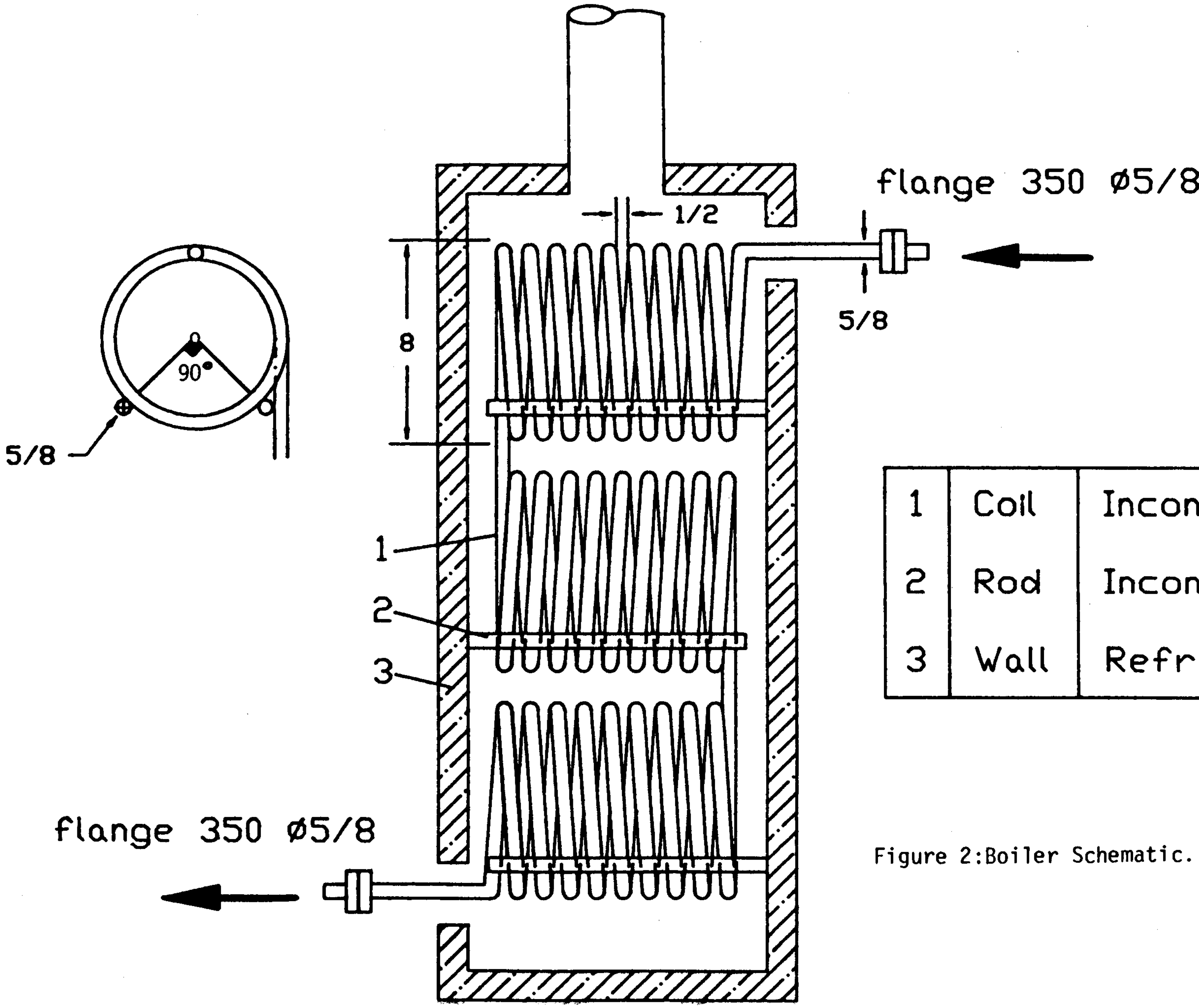


Figure 1: Pilot Plant For Processing CNSL



1	Coil	Inconel 600
2	Rod	Inconel 600
3	Wall	Refractory

Figure 2:Boiler Schematic.

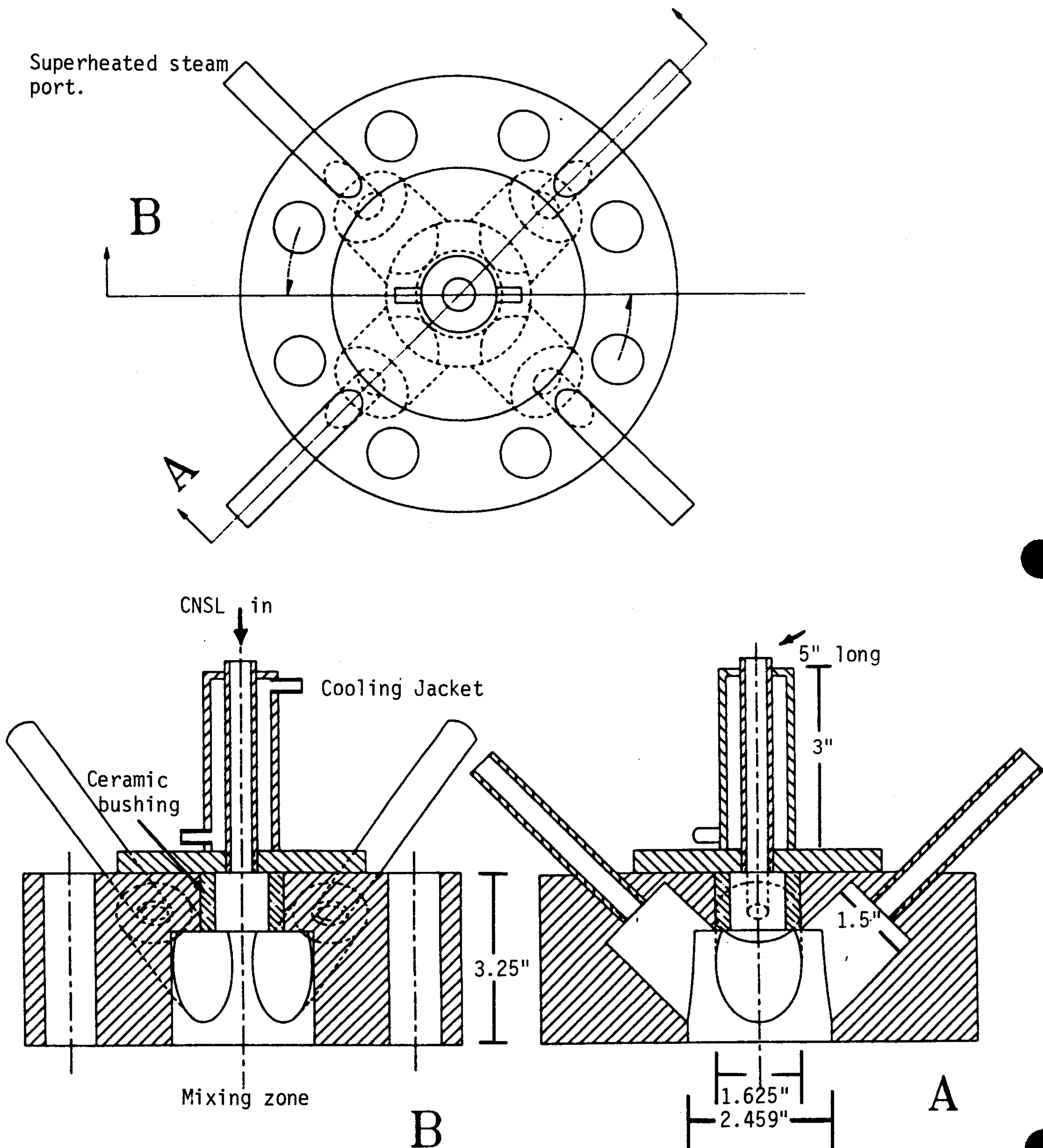


Figure 3: Reactor Schematic.
(Reactor Head)

Products and reactants exiting from the reactor then pass through a quencher hopper (Figure 4), built out of stainless steel, where they are immediately quenched by water injection to stop the cracking reaction as quickly as possible. In the quencher-hopper, liquid water is injected through four nozzles, producing atomized water jets which hit the incoming stream of gaseous products and reactants are deflected towards the side of the hopper by a conical surface. A series of 4 thermocouples located in different radial positions inside the hopper are used to record the quenching temperatures.

The gaseous mixture of products and reactants exiting the hopper are sent to a condensation unit, then to a gas separator allowing the separation of liquid and gaseous phases. Liquid is withdrawn from the bottom and gases are sent to special gas bags for analysis.

Proposed Operational Procedure of the Pilot Plant

The following operational procedure, already reviewed with UEM researchers, was set to operate the proposed pilot plant in Mozambique:

Start-up

- The unit is heated to the desired temperature in the various sections by superheated steam generated in the boiler. A series of 7 thermocouples are used to monitor the temperatures of the pilot plant. Once the set points are reached, the hopper is opened, drained and closed.

- Superheated steam flowrate is set at the desired value.

- CNSL and Quench pumps are turned on simultaneously.

- At this stage, the experimental run is initiated.

- This operation continues for about 20 minutes or until steady state operation of the pilot plant is established. Once it is reached, this is considered to be the start time of the sampling period (time = 0), all liquids collected in the condenser in the 20 minute start up period are drained.

- The operation of the pilot plant is allowed to continue for another 15 to 20 minutes after steady state is reached. All the liquid and gases are collected for the duration of the sampling period. These amounts are used to assess the mass balance of the experimental run and simultaneously the operation of the pilot plant. Samples of the liquid and gas portions are then analysed to identify the main reaction products by gas chromatography.

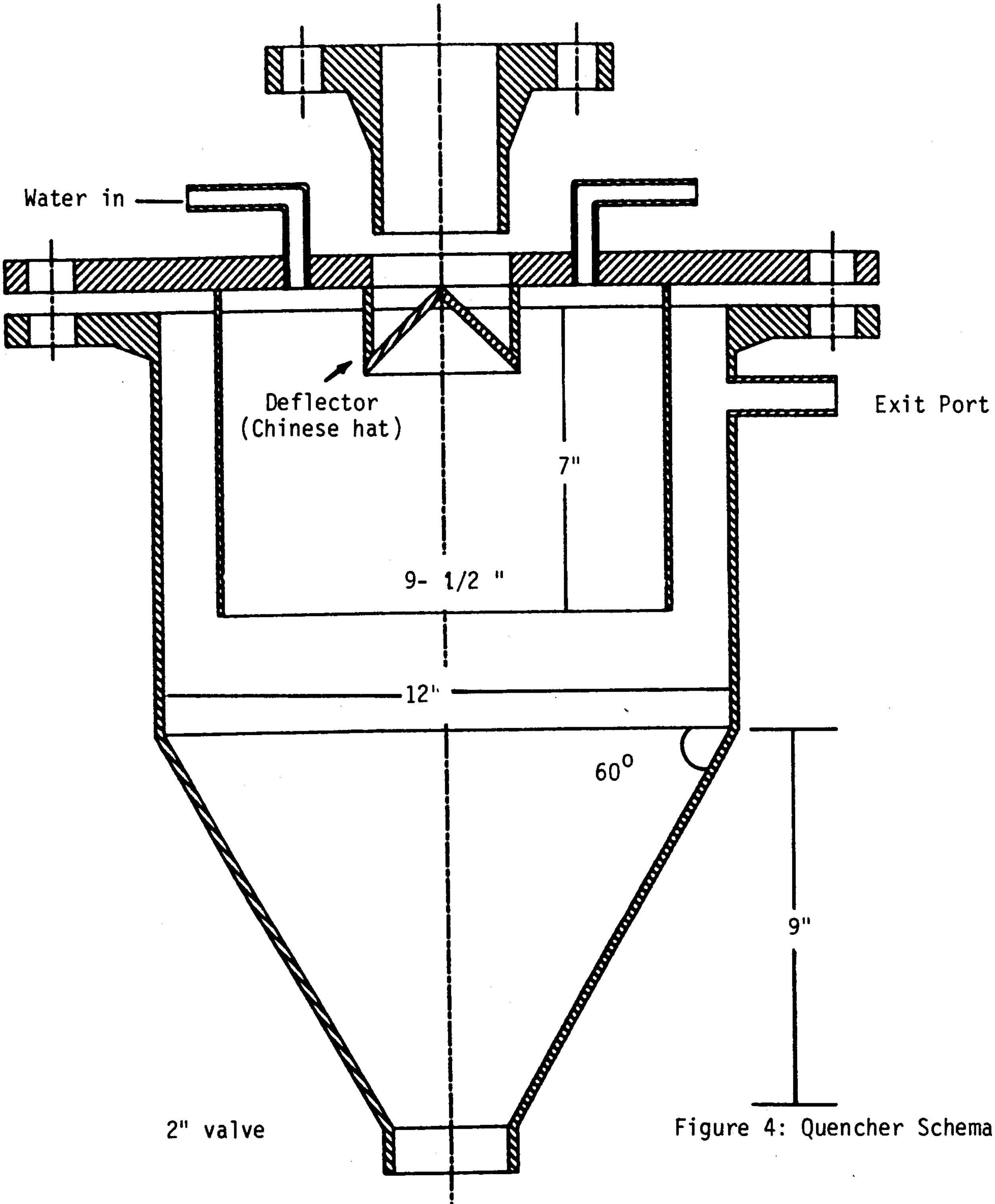


Figure 4: Quencher Schematic.

Shut down or preparation for another run

- At the selected time for ending the run, CNSL flow is shifted to water, superheated steam and quenching lines are kept on in order to flush the lines in the system and for preventing the nozzles from plugging. This phase of the operation must continue for a period not less than 60 minutes or until UEM researchers are certain that all nozzles in the system are working and all equipments are in good working order, i.e. system is clean.

- The pilot plant can be either switched off or prepared for another run.

Mass Balance and Conversion Calculations.

Furthermore, a procedure is also suggested to the researchers from the University Eduardo Mondlane (UEM) for mass conversion calculations. This allows the direct assessment and performance of the unit on a systematic basis.

The following is the recommended procedure to perform overall mass balance calculations around the pilot plant unit.

Mass of CNSL fed will be compared with mass of CNSL recovered. Note that this analysis will be performed with data collected during the sampling period after the system reached steady state.

A. Mass of CNSL (g) = Feedstock flow (g/s) × Run time

B. Mass of CNSL recovered (g) =
Liquid sample collected in separator (g) +
Gas sample collected in gas bag (g) +
Tar and coke sample collected in separator.

Regarding the various fractions involved in the overall mass balances the following should be clarified:

- The mass of liquid CNSL collected will be directly weighed.

- The mass of gaseous CNSL collected in the gas bag, after evacuation of the same into a bottle, where vacuum was previously produced, will be evaluated as follows:

Gas mass = $MW_{AVGgases} \Delta P_{bottle} V_{bottle} / R T$

where ΔP_{bottle} is the measured pressure change in the bottle due to the evacuation of the gas bag, V_{bottle} is the known volume of the bottle, $MW_{AVGgases}$ is the average molecular weight of the CNSL gaseous mixture (assessed from gas chromatograph analysis - TCD), R is the universal gas constant and T is room temperature.

- Tar will be assessed by periodic washing and or inspection of the equipments.

Regarding the important conversion parameter the following calculation was performed for each run:

$$x_{Feedstock} = 100 \text{ x Products (g) / Mass of feedstock fed (g)}$$

$$= [M_{gas} + M_{liquid} (1 - Y_{feedstock}) + \text{Coke}] / M_{feedstock\ fed}$$

where $Y_{feedstock}$ is the proportion of feedstock found in the liquid sample (assessed by gas chromatography analysis - FID).

<u>Construction of Components</u>

Researchers at CREC are involved in handling a number of projects that are dealing with reactors operated at high temperatures (around 900°C). These novel reactors were designed by CREC researchers and fabricated by the Machine Shop of the University of Western Ontario. As well, they are made out of inconel, the metal of choice for the reactor of the Mozambican pilot plant.

Thus, it was decided to construct the following components in Canada: the reactor, the quencher and coils for the furnace. The rest of the pilot plant is to be handled locally in Maputo and it consists of the following items: cooler, steel structure, separator and decanters.

In Canada, the progress of the construction of the components of the above project is progressing as follows:

Reactor:

This unit was designed and manufactured following the specifications given in Figure 1. It is now packed and ready for shipment to Mozambique.

The UWO-Mechanical Shop completed the feeding section of the reactor in early 1992 (reactor upper section). The Inconel tube for the reactor body was well received and the two reactor sections were machined. Inconel flanges were welded to the two reactor sections. Inserts for thermocouples were manufactured. All the components for injectors distribution of steam and cooling jacket for CSNL were already welded. The reactor was manufactured out of inconel and subjected to system pressure tests.

Quencher:

This unit was manufactured following the specifications of Figure 4. It is now packed and ready for shipment to Mozambique.
This unit, to be subjected to lower temperatures (in the 300 °C range), was manufactured out of stainless steel.

Furnace:

Three sections of the coil were manufactured out of Inconel and welded by the Mechanical Shop-UWO (An additional one was manufactured as spare if needed). The coil and Inconel supports has been completed, packed and ready for shipment to Mozambique. At this stage we are in the process of further investigating the purchase of the oil burners in order to provide the proper oil combustion flows and to make sure that they are built with the proper materials.

All of these sections were thoroughly tested and inspected by competent personnel of the UWO Mechanical Shop.

On Thursday November 21, 1991, the state secretary of Cashew, Dr. Juliano Maria Saranga and Dr. Jose Jamisse visited the University of Western Ontario and toured the premises of CREC. The progress of the project was discussed and they were given photographs of the units under construction by the Mechanical Shop of the University of Western Ontario. As well, as that time, we were still waiting for instructions from Mozambique regarding the proper method of shipping the computer and the fax machine in order to establish direct contact with the researchers from the University Eduardo Mondlane.

Ordering Components

- Pumps: The 4 pumps required were purchased and delivered to UWO. The three positive displacement piston pumps were acquired in dual voltage. Please note that the self priming pump was only available in 115 volts. Electrical work related to these items should be carried out locally in Mozambique (plugs, wiring etc).
  These units have been completed, packed and ready for shipment.

- Probes with Sheet type K: Ordered and delivered to UWO.
  These units have been completed, packed and are ready for shipment.

- Thermocouple Switch Boxes: Ordered and delivered to UWO.
  This unit has been completed, packed and ready for shipment.

- Gas sampling bags and syringes for gas and liquid samples: Ordered and delivered to UWO. This unit has been completed, packed and ready for shipment.

- Nozzles for the reactor and quenchers: 12 nozzles were purchased which is going to provide 4 extra nozzles. A sonic bath was also purchased to help with the eventually

required cleaning of nozzles (ordered and delivered to UWO).
These units have been completed, packed and ready for shipment.

- Burners: Still not ordered. After reviewing all the collected materials, it was found that the proposed FAFC oil burners were not suitable for this kind of operation because they are equipped with a relight type control that causes an explosion hazard when used in a combustion chamber that is equipped with refractory liner (brick, ceramic or castable refractory materials). Brick is to be used to built the proposed unit in Mozambique. Currently we are seeking additional information regarding other types of burners.

- Insulations materials were completed, packed and ready for shipment..

- Pressure gages ordered and delivered to UWO. This unit has been completed, packed and ready for shipment.

- Fax machine & Computer: Both units were purchased, packed and sent to Mozambique by cargo air on January 21, 1992, the Fax machine and the computer were shipped by Air Canada as per Dr. Da Maia instructions to our customs and traffic officer (Airwaybill Number 59344261). We are still awaiting access to a new fax number to establish direct contact with researchers at Mozambique and determine additional needs for support equipments related to the set-up and the progress of this project.

- Miscellaneous
  As needed, other items such as extra valves, compression fittings, copper tubing etc... that related to this project are being ordered and stored at UWO. It should be mentionned here that most fitting related to the pilot plant were designed in a way that facilitate integration and easy installation of various parts and components of the pilot plant in Mozambique. This will reduce the cost of operation and maintenance of this unit.

Updated Timetable

This project has been delayed in part because direct contacts between the parties involved are somewhat slow and not yet fully established. Direct line of communication, via the fax machine, will help considerably day-to-day interactions during the set-up and operation of the pilot plant unit. In this way, the operation of the unit could be closely monitored by both parties UEM and CREC.

In this context, and considering all initial objectives could be achieved, CREC would like to request an extension of 10 months to the original proposed timetable (from March 1 to December 31, 1993). It has to be stressed that this extension, in no way, will affect the budget assigned to CREC. On the other hand, it will help to coordinate in a positive way the progress of the project that so far has met good results and positive expectations.

The new proposed timetable can be summarized as follows:

1 - Shipping of the pilot plant (October - November, 1992).

2 - Delivery and assembly in Mozambique (December - January, 1993).

3 - Trial Tests to debug the system and become familiar with its operation (February - April, 1993).

4 - Visit to Mozambique (Dr. H. de Lasa and Mr. S. Afara) to review the progress and the debugging of the pilot plant as needed (May, 1993).

5 - Optimization and extended runs (June - September, 1993).

6 - Visit to Mozambique (Dr. M. A. Bergougnou) (August, 1993).

Conclusions:

The success of the preliminary study to characterize the CNSL and to obtain some estimates of the product yield (light gases, phenol, tar) as a function of temperature and residence time helped to demonstrate the feasibility of this technology.

A similar more complex pilot plant of the same material of construction "inconel" is still in operation at CREC since 1990. In this unit, experimental runs dealing with catalytic craking of model compounds, with catalyst used as heat carrier, were performed. Even after 200 experimental runs, the pilot plant has not shown or required any major modification to its design aspects. The researchers at CREC, involved in this project, are very proud of their safety record and eager to pass their experience in dealing with day-to-day operation of the pilot plant to the researchers at UEM.

Once direct communications are established between UEM and CREC, by means of the Fax system, matters requiring immediate attention such as the set-up of the pilot plant and day-to-day operation of the unit will be handled in a faster fashion.

It is our view that an extension of 10 months, will improve significantly the success of this project and the final outcome.

In summary, the equipments to be manufactured in Canada, as well as other support materials are listed in the enclosed table. All the materials that were crated are ready for shipments. The shipping department at the University of Western Ontario has already contacted Dr. Da Maia in Mozambique. They are awaiting instructions from Mozambique related to proper routes to be used to send the crate. In addition, oil burners will be rushed by air cargo as soon as design aspects are finalized with UWO Mechanical Shop.

Shipping List

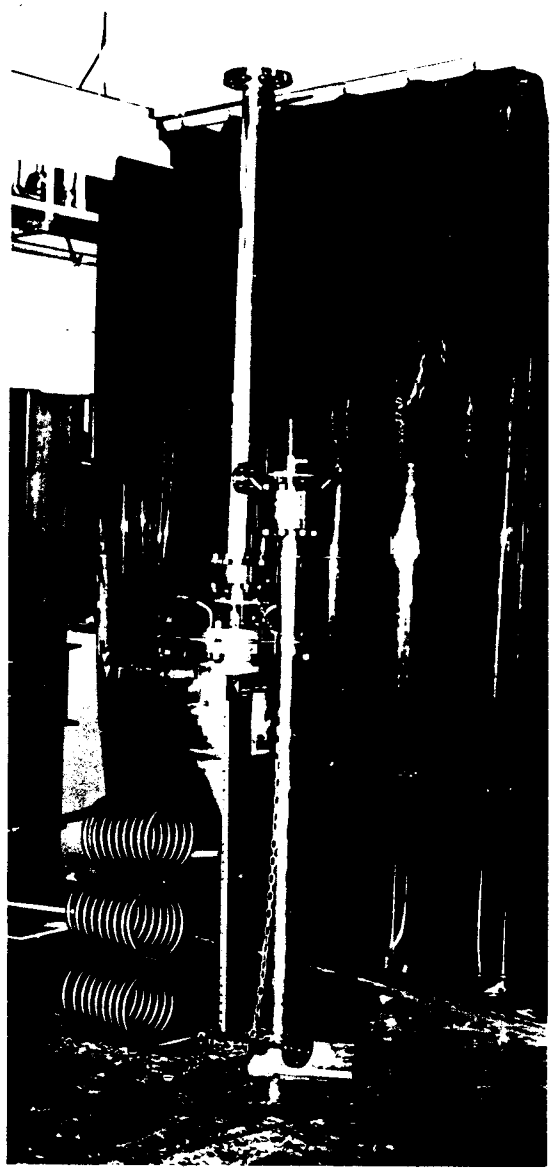
#	CATALOG #	Manufacturer	Description
1	L-07080-50	Cole Parmer	Pump, self prime
1	L-07128-50	Cole Parmer	Pump, meter
1	L-07128-15	Cole Parmer	Pump, meter
1	L-07128-20	Cole Parmer	Pump, meter
2		Calibrated Inst.	Sampling Bags
1	H80380	Chroma. Spec.	GC Syringes
2	H81620	Chroma. Spec.	GC Syringes
2	H90022	Chroma. Spec.	GC Syringes
16		Ind. Mech.	Nozzles
1	C6431-100	Can Lab.	Sonic Bath & Tray
2		Can Lab.	Nalgene Tank
1		Can Lab.	Spigot
4		Carborundum	Insulation
18		Omega	Thermocouples
1		Omega	Therm. Wire
1		Omega	Temp. Ind.
1		Omega	Switch

* Miscelleneous compression and pipe fittings in brass, stainless steel, copper to connect all lines and pressure gages.

* Reactor from Inconel manufactured by UWO-Mechanical shop
  2 pipes from Inconel manufactured by UWO-Mechanical shop
  1 stainless steel hopper manufactured by UWO-Mechanical shop
  1 hopper holder from carbon steel manufactured by UWO-Mechanical shop

* Oil burners not purchased yet.

* Crate Dimensions: 5' x 3' x 3'3"
  weight: N/A



1 Reactor head from Inconel manufactured by UWO-Mechanical shop
2 pipes from Inconel manufactured by UWO-Mechanical shop
1 stainless steel hopper manufactured by UWO-Mechanical shop
1 hopper holder from carbon steel manufactured by UWO-Mechanical shop
1 coil from Inconel manufactured by UWO-Mechanical shop